

NASA Technical Memorandum 107437

MS212–A Homogeneous Sputtered Solid Lubricant Coating for Use to 800 °C

Harold E. Sliney
Lewis Research Center
Cleveland, Ohio

William J. Waters and Richard Soltis
OMNI Corporation
Cleveland, Ohio

May 1997



National Aeronautics and
Space Administration

MS212-A HOMOGENEOUS SPUTTERED SOLID LUBRICANT COATING FOR USE TO 800 °C

Harold E. Sliney*
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

William J. Waters and Richard Soltis
OMNI Corporation
Lewis Research Center
Cleveland, Ohio 44135

INTRODUCTION

A composite material, invented at the Lewis Research Center of NASA (refs. 1 and 2), is self-lubricating at high temperatures to about 800 °C. It contains by weight percent: 70 metal-bonded chromium carbide, 15 silver, and 15 barium fluoride/calcium fluoride eutectic. This composition has been used as a plasma-sprayed coating (refs. 3 and 4). It has also been fabricated into powder metallurgy parts by pressureless sintering (refs. 5 to 7), hot pressing (ref. 7), and extrusion (ref. 8). The plasma sprayed coatings are designated as PS212, the powder metallurgy products as PM212.

All of these preparation methods produce useful bearing surfaces. However, where the application calls for a lubricant coating that is thinner than the customary plasma spray coating or for which a powder metallurgy bearing is not suitable, a thin sputtered coating may provide a solution. Also, sputtered coatings may be used in the as-applied condition, while products made by the other techniques require machining to final dimensions. The coatings studied in this program were applied by the physical vapor deposition (PVD) process of magnetron sputtering. We refer to them as MS212 coatings.

Sputtering deposition parameters were varied empirically until surface chemical analyses showed that a satisfactory composition had been achieved. The friction and wear properties of the coating were evaluated in pin on disk sliding tests. Tests were performed at room temperature on coated aluminum and from room temperature to 800 °C on coated Inconel X-750. Baseline tests of the uncoated metals were performed for comparison.

MATERIALS

Chemical Composition of MS212

The nominal, desired composition of MS212 by weight percent is:

70 metal bonded chromium carbide
15 silver
15 barium fluoride/calcium fluoride eutectic

The coating materials are sputtered from a sintered primary target of this chemical composition and a secondary target of silver. Chemical analyses in a scanning electron microscope (SEM) by x-ray energy dispersive spectroscopy (EDS) showed that all chemical elements in the target were present in the deposited coating. The desired quantitative composition was empirically achieved by systematically adjusting the sputtering parameters and by using a secondary target of silver. The secondary target was needed because silver is easily resputtered from the coating during the deposition process. Parameters that produced adherent coatings in this program are given in table I.

The EDS spectrum for MS212 is shown in figure 1. The molecular composition was studied using x-ray photoelectron spectroscopy (XPS). This technique measures the binding energies of the atomic species in the sample

*Distinguished Research Associate.

which in turn identify the compounds that are present. All compounds in the coating were identified as those in the target within the resolving power of the instrument.

Counterface and Substrate Materials

The sliding counterface materials are hemispherically-tipped pins of aluminum oxide or of the cobalt based alloy Stellite 6B. The coated metal disks are made of a soft aluminum alloy or the nickel base alloy Inconel X-750. The Rockwell hardness of alloys 6B and Inconel X-750 is RCH 38. Titanium 6Al-4V and the nickel alloy TAZ 8A were also coated for scratch test evaluation.

TEST PROCEDURES

Scratch Hardness

Scratch hardness tests were performed on the MS212-coated aluminum, titanium 6-4, and the nickel based alloys, Inconel X-750 and TAZ 8A to study their crack resistance when subjected to a dynamic load condition. The scratch hardness tester moves a diamond stylus, which has a 200 μm tip radius, across the coating surface at a velocity of 100 mm/min and a stylus loading rate of 100 gms/min. This generates a tapered scratch. The output of an acoustic emission sensor is plotted versus stylus load. A sharp increase in acoustic emission indicates the load at which the first cracks are initiated. Friction force is also plotted. Friction increases linearly with load until the stylus breaks through the coating at which point a sharp jump in friction is observed. Therefore, the critical load for crack initiation is mutually confirmed by acoustic emission and friction force measurements.

Friction and wear tests

These tests are conducted using the high temperature pin on disk tribometer shown in figure 2. The disk specimens in this study are MS212-coated and baseline uncoated aluminum and the nickel based turbine alloy, Inconel X-750. The pins are the cobalt base alloy Stellite 6B with a hemispherical tip radius of 4.76 mm and aluminum oxide with a tip radius of 25.4 mm. The sliding contact geometry therefore is essentially that of a ball on a flat. The pin generates a 50 mm diameter wear track on the disk. Friction force is continuously measured and recorded during the tests.

As wear occurs, a circular wear scar is formed on the tip of the pin and a grooved wear track is formed on the disk. Pin wear is quantified post-test by measuring the wear scar diameter on a calibrated photomicrograph of the pin, then calculating the volume of the spherical segment that has worn away. Disk wear is measured as follows: A stylus profilometer traces and computes the cross sectional areas in several locations of the wear track, the average of these areas is then multiplied by the track circumference to give the wear volume.

Wear factors, k , are then calculated by dividing the wear volume by the load and the total distance of sliding. The equation and units are:

$$k = \text{mm}^3/\text{N}\cdot\text{m}$$

DISCUSSION

Microstructure

As previously mentioned, all of the components of the sintered sputter target are present in the MS212 coating. Figure 3(a) is an SEM of the surface of sputtered MS212. The coating is seen to be homogeneous with no distinguishable grain boundaries or phase segregation. In contrast, the diamond ground surface of sintered PM212 shown in figure 3(b) clearly shows segregation of fluorides and silver (white areas) from the darker bonded carbide phase.

The cross section microstructures of sintered PM212 shown in figure 4 are very coarse with relatively large areas of segregated phases. While the coarse microstructure has proved to be acceptable in sliding contact journal bearing (ref. 9), applications requiring thin coatings that follow the topography of the substrate surface are expected to require a more homogeneous microstructure. Further, PS212 and PM212 must be diamond ground to produce an acceptable bearing surface while MS212 is usable as-coated.

Figure 5(a) is presented to illustrate how MS 212 follows the microtopography of a scratched surface. Faithful replication however, requires a reasonably smooth substrate surface. Figure 5(b) illustrates the nodular growth of MS212 on a glass-peened surface

Load Limits

The tapered scratches formed during scratch tests on MS212-coated aluminum and titanium 6-4 are shown in figures 6(a) and (b). Friction force (F) and acoustic emission (AE) at a uniformly increasing load are shown in figure 6(c). At a critical load on the stylus, the coating cracks and spalls. The critical load for crack initiation is of course a function of substrate deformation under load. As expected, the limiting load for crack initiation is higher on the titanium alloy than on the softer aluminum.

The Vickers diamond point hardness (DPH) of uncoated and MS212-coated aluminum and titanium 6-4 are shown as a function of stylus load in figures 7(a) and (b). DPH as a function of stylus load for two MS212-coated nickel alloys, Inconel X-750, a similar nickel alloy, TAZ8A, and uncoated TAZ8A are shown in figure 7(c). With the exception of an unexplained anomalously low hardness of coated Ti 6-4 at an indenter load of 50 gms, the hardness numbers at indenter loads of 50 and 100 gms for all four coated metals are $600 \pm 80 \text{ kg/mm}^2$, presumably the coating hardness. As load increases, the DPH numbers approach those of the substrate metals. Note that the DPH numbers for both nickel base alloys decrease only slightly with stylus load at loads up to at least 500 gms.

Friction and Wear

Aluminum substrate.—The coefficients of friction for 6B pins sliding at room temperature under a load of 1N on uncoated aluminum and on aluminum coated with 20 μm thick MS212 are given in figure 8. At a sliding velocity of 0.27 m/s, friction coefficients are 1.0 to 1.4 for the baseline uncoated aluminum. Friction coefficients are markedly lower for the coated case, usually at about 0.27 ± 0.03 for sliding velocities of 0.27, 0.68, and 1.35 m/s, although values as high as 0.40 have been observed.

Figure 9 gives the friction coefficients at room temperature at a sliding velocity of 0.34 m/s for aluminum oxide pins sliding on aluminum disks coated with 20 μm of MS212. Tests were conducted at 1, 2, and 4.9N loads. Friction coefficients were a very steady 0.35 at 1N, about 0.45 at 2N, and 0.5 to 0.6 at 4.9N. As expected, the substrate deformation at the higher loads caused coating failure. The results show that the coating effectively lubricates a concentrated contact on aluminum at light loads (low contact pressures), and suggests that the coatings may be more durable in conforming contacts such as a flat on flat or cylindrical bearing where contact areas are much larger and contact pressures are comparatively low.

Inconel substrate.—The friction coefficients of Stellite 6B pins sliding on uncoated and MS212-coated Inconel X-750 at room temperature and at 350 °C are given in figure 10. At room temperature, friction coefficients are reduced from 0.60 ± 0.07 to 0.28 ± 0.02 by the MS 212 coating. Silver coatings are also effective at room temperature, but do not reduce friction as much as MS212. At 350 °C, MS212 reduces friction coefficients from 0.51 ± 0.05 for the baseline unlubricated metals to 0.25 ± 0.02 .

Figures 11(a) and (b) give the friction and wear under a 5N load from 25 to 800 °C for the case of aluminum oxide pins sliding on uncoated and MS212-coated Inconel X-750. Two coating thicknesses were tested, 1.4 and 20 μm . The coatings reduced friction compared to the baseline at all test temperatures. Pin wear against the 20 μm thick MS212 coatings was an order of magnitude lower than pin wear against uncoated Inconel X-750 at 25 and 350 °C. Pin wear coefficients are low (10^{-7} to $10^{-6} \text{ mm}^3/\text{Nm}$) at 600 and 800 °C against both the baseline and the coated disks. The low pin wear in spite of higher friction against uncoated Inconel X-750 at these high temperatures is attributable to the natural oxides that form on this alloy above about 500 °C in an oxidizing air environment. The lubricious nature of oxides on nickel-chromium super alloys at high temperatures is well documented, (e.g., ref. 10).

CONCLUSIONS

Aluminum

1. At room temperature, sputtered MS212 coatings on the flat surfaces of aluminum disks markedly improve the friction and wear during sliding under light loads against hemispherically-tipped pins of the cobalt alloy Stellite 6B or aluminum oxide ceramic. For example the friction coefficient of Stellite 6B against uncoated aluminum is very high at 1.2 ± 0.2 . By comparison, the friction coefficient for 6B or aluminum oxide sliding on MS212-coated aluminum is typically 0.35 ± 0.05 .
2. At light loads, the coating also prevents the severe galling wear typical of aluminum in sliding contacts.
3. Effective lubrication of aluminum by MS212 coatings is load-limited. Aluminum is considerably softer than the coating material. At high loads, plastic deformation of the aluminum substrate causes the coating to crack and spall from the surface of the aluminum.

Inconel X-750

1. At room temperature, friction coefficients are 0.60 ± 0.07 for Stellite 6B pins sliding on uncoated Inconel-X 750. Sputter coating the disks with MS212 reduces the friction coefficients to 0.25 ± 0.03 . At 350 °C, friction coefficients are 0.51 ± 0.09 uncoated versus 0.25 ± 0.03 coated.
2. MS 212 coatings also reduce the friction coefficient of ceramic (alumina) pins sliding on Inconel X-750 disks at all temperatures from 25 to 800 °C. For example: At room temperature, friction coefficients are 0.67 ± 0.07 uncoated versus 0.45 ± 0.10 coated. At 800 °C, friction coefficients are 0.36 ± 0.04 uncoated versus 0.29 ± 0.04 coated.
3. At 25 and 350 °C, the wear rates of alumina pins sliding on Inconel-X 750 coated with 20 μm thick MS212 are about one-tenth the wear rates for the uncoated metal. At 600 and 800 °C, wear rates are low for both the MS212-coated and the uncoated baseline cases. At these high temperatures, the lubricious oxides that form on the disks are effective in wear protection, but not as effective as MS212 in reducing friction.
4. As with any hard coating on a softer substrate, lubrication with MS212 is load-limited in concentrated contacts such as the pin on disk configuration used in our tests. Scratch tests at progressively higher loads show, as might be expected, that the crack initiation load for the MS212 coating in a concentrated contact is a function of the deformation properties of the substrate. Therefore, although MS212 lubricates soft metal such as aluminum in lightly-loaded hemisphere on disk tests, these coatings are shown to have much higher load carrying capability on a harder, higher modulus substrate such as Inconel X-750.

ACKNOWLEDGMENTS

This is to acknowledge the assistance of the Commercial Technology Office of this Center in supporting part of the research reported in this paper. We also acknowledge the contribution of Michael S. Bogdanski who performed the wear tests described herein while a student aide from Case/Western Reserve University.

REFERENCES

1. Sliney, H.E.: "Carbide, Fluoride, Silver Self-Lubricating Composite," U.S. Patent 4,728,448, Mar. 1, 1988.
2. Sliney, H.E. and DellaCorte, C.: "Method of Making Carbide/Fluoride/Silver Composites," U.S. Patent 5,034,187, Jul. 23, 1991.
3. Della Corte, C. and Sliney, H.E.: "Composition Optimization of Self-Lubricating Chromium Carbide-Based Composite Coatings for Use to 760 °C," ASLE Trans. 30, 1, pp. 77-83, Jan, 1987.
4. Sliney, H.E.: "A New Chromium Carbide-Based Coating for Use to 900 °C with Particular Reference to the Stirling Engine," J. Vac. Soc. Technology, A4, 6, pp. 2629-2632, Nov.-Dec. 1986.
5. DellaCorte, C. And Sliney, H.E.: "Tribological Properties of PM212: A High-Temperature, Self-Lubricating Powder Metallurgy Composite," Lubr. Engr. 47, 4, pp. 298-303, 1991.

6. Sliney, H.E., DellaCorte, C., and Lukaszewicz, V.: "The Tribology of PS212 Coatings and PM212 Composites for the Lubrication of Titanium 6-4-Al Components of a Stirling Engine Space Power System," STLE Tribology Transactions, 38, 3, pp. 497-506, 1995.
7. Bogdanski, M.S., Sliney, H.E., and DellaCorte, C.: "The Effect of Processing and Compositional Changes on the Tribology of PM212 in Air," Lubr. Engr., 58, 8, pp. 675-683, 1995.
8. Waters, W.J., Sliney, H.E., Soltis, R.F., and Bemis, K.: "Properties of Extruded PS212 Type Self-Lubricating Materials." Proceedings of NASA Tech. 2002 Conference, Balt, MD, Dec. 1992. Published in NASA CP-3109, 1, pp. 41-55, Feb. 1993.
9. Sliney, H.E.: "Sliding Contact Bearings for Service to 700 °C," NASA TM-107307, Sept. 1996.
10. Laskowski, J.A., and DellaCorte, C.: "Friction and Wear Characteristics of Candidate Foil Bearing materials from 25 to 800 °C," STLE Jour. of Lubrication Engr., 52, 8, pp. 605-612, Aug., 1996.

TABLE I.—SPUTTER PARAMETERS^a

Technique.....	RF magnetron sputtering
Atmosphere.....	8×10^{-3} torr Argon
Target preparation	Sputter-cleaned for 5 min immediately before deposition
Targets	Sintered PM212 and silver
Target bias, V	145
Power level, kW	0.5 or 1.0
Deposition rates	270 Å/min at 500 W 625 Å/min at 1 kW

^aOptimum sputter parameters will vary from one sputter system to another. The above parameters are those which produced adherent coatings on Inconel X-750 in this program.

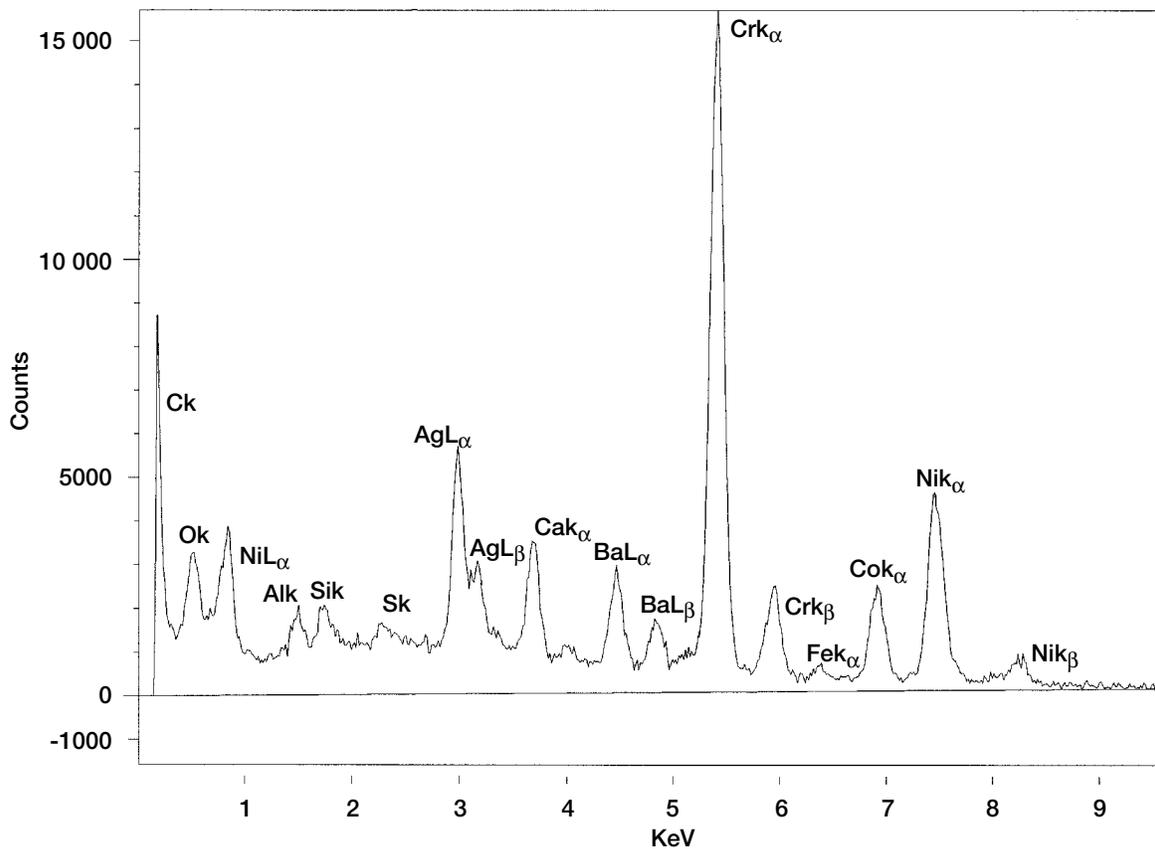


Figure 1.—Energy dispersive spectrum (EDS) of sputtered MS212.

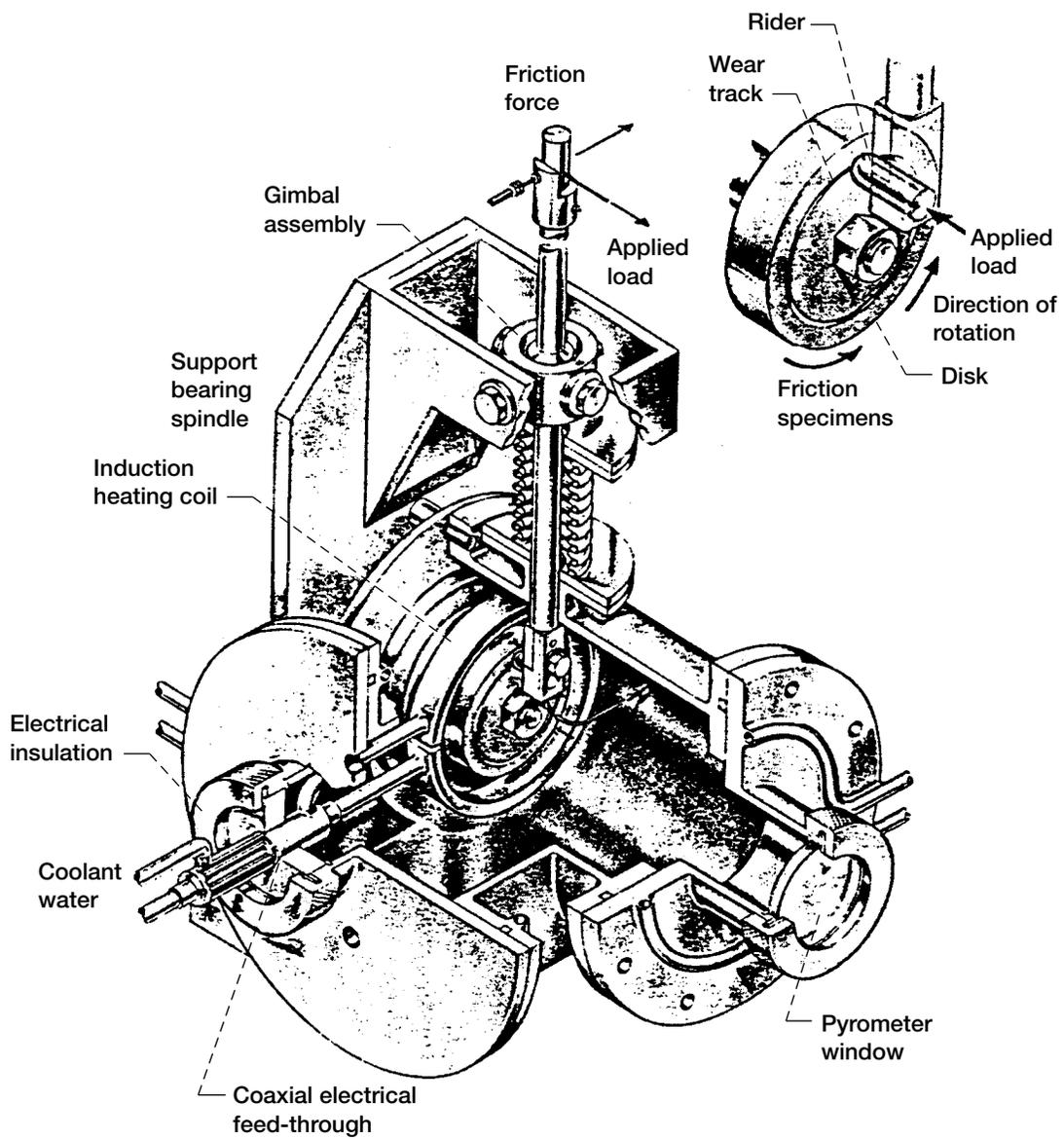


Figure 2.—Pin on disk friction and wear test machine.

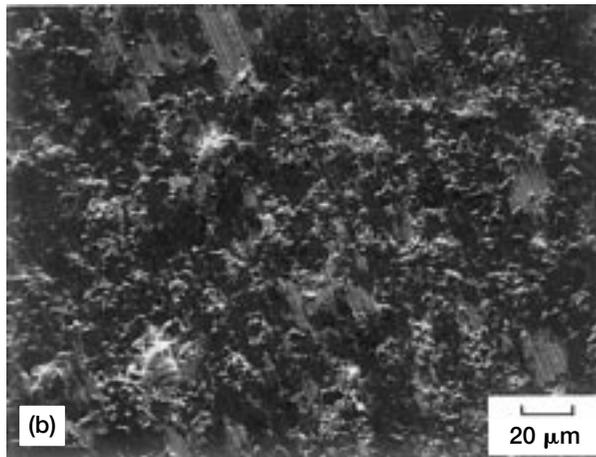
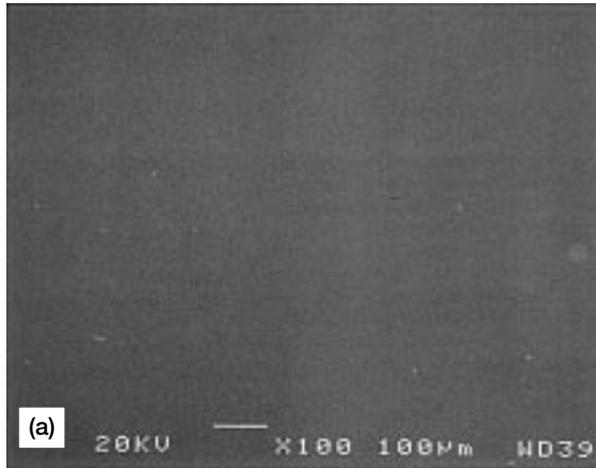
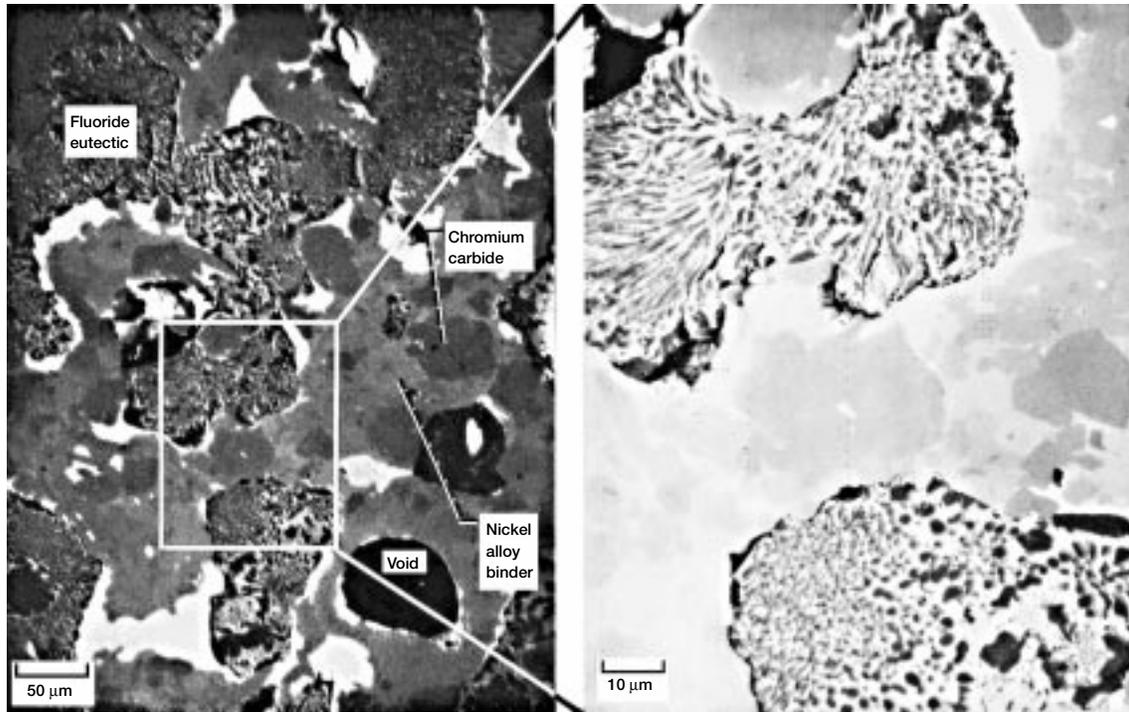
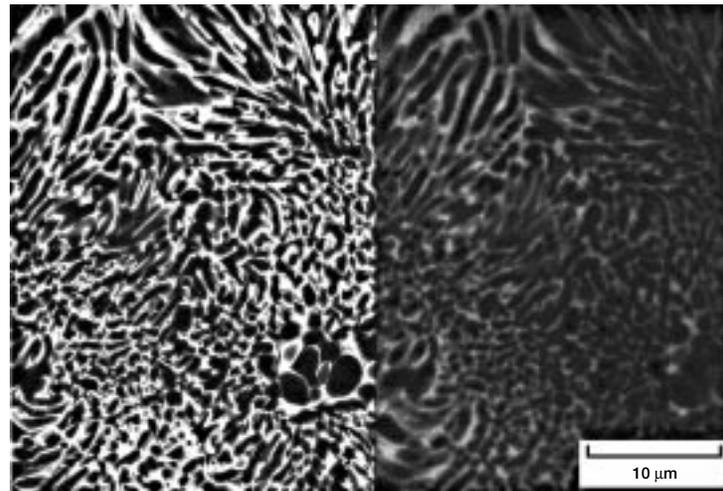


Figure 3.—Surface microstructures. (a) Magnetron-sputtered MS212 on a polished substrate. (b) Diamond ground surface of PM212.



Distribution of components in PM212 microstructure.



Distribution of components in PM212 microstructure.

Figure 4.—Cross-section microstructure of PM212 by back scatter electron microscopy.

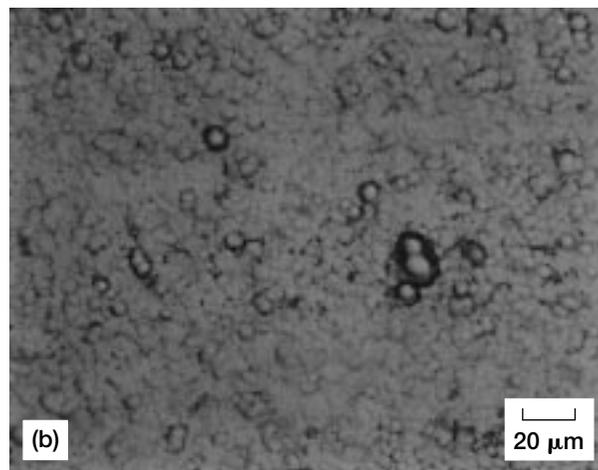
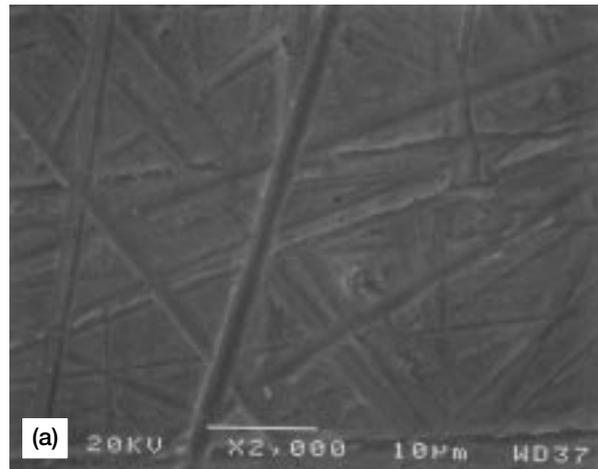


Figure 5.—Micrographs of; (a) MS212 on scratched substrate showing accurate replication of substrate surface topography. (b) Nodular growth of MS212 on a glass-peened substrate.

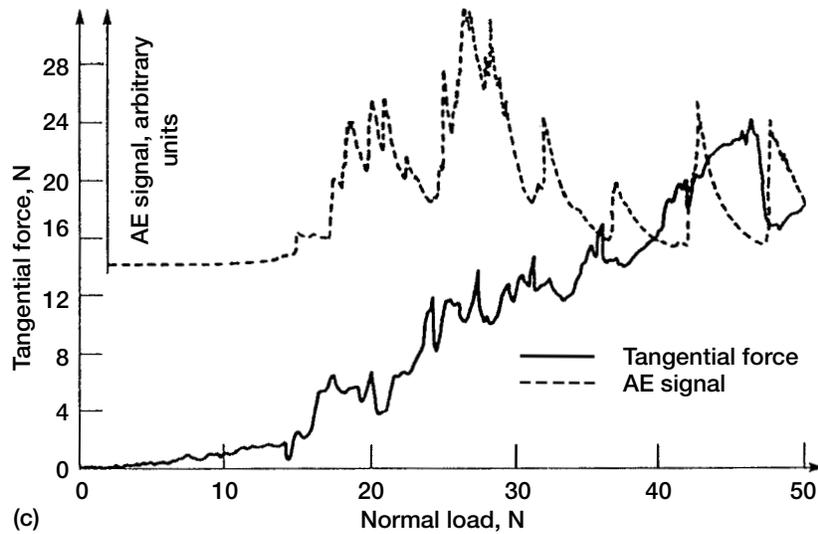
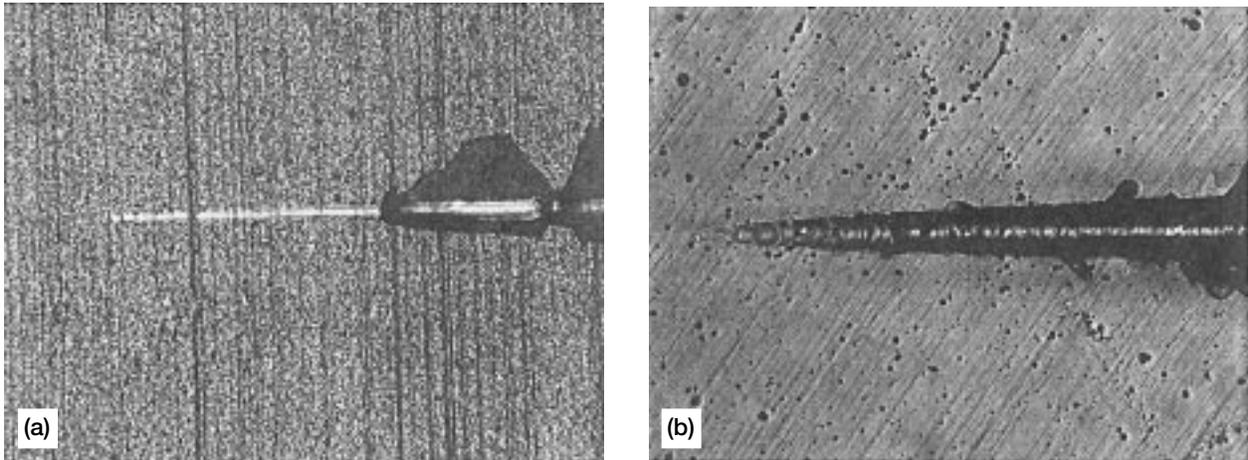


Figure 6.—Scratch tests impressions. (a) Titanium 6-4. (b) Aluminum. (c) Friction force (F) and acoustic emission (AE) versus stylus load for MS212-coated Ti 6-4.

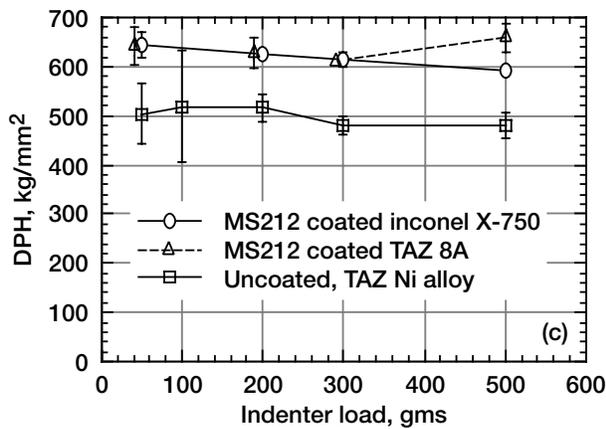
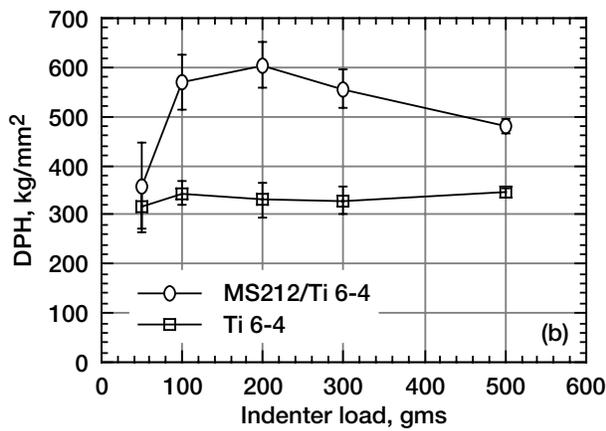
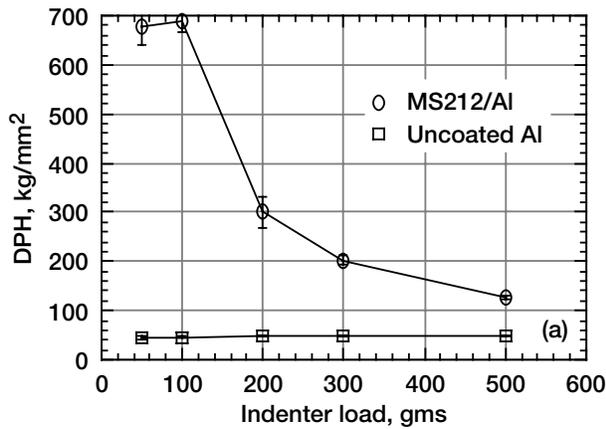


Figure 7.—Diamond point hardness of MS212-coated metals. (a) Effect of 20 micron thick MS212 coating on Vickers superficial hardness of aluminum. (b) Titanium 6Al-4V. (c) Nickel super alloys.

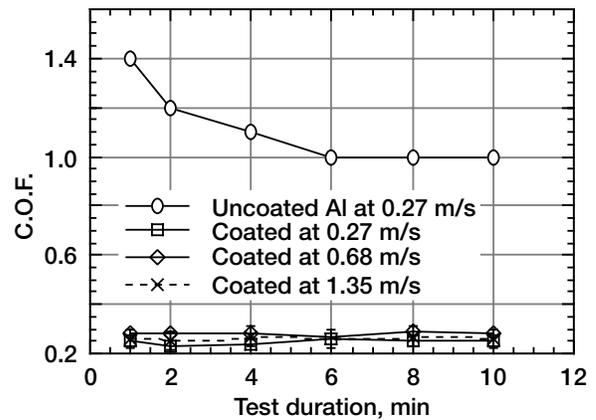


Figure 8.—Coefficient of friction (COF) at room temperature for cobalt alloy 6B sliding on uncoated aluminum and aluminum coated with 20 micron thick MS212.

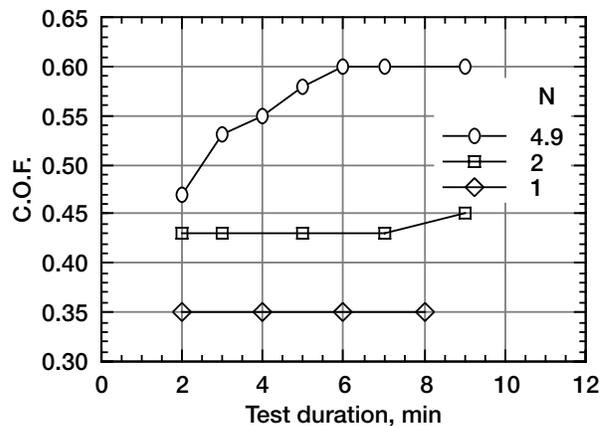


Figure 9.—COF for alumina pins sliding against 20 microns thick MS212 on aluminum at various pin loads.

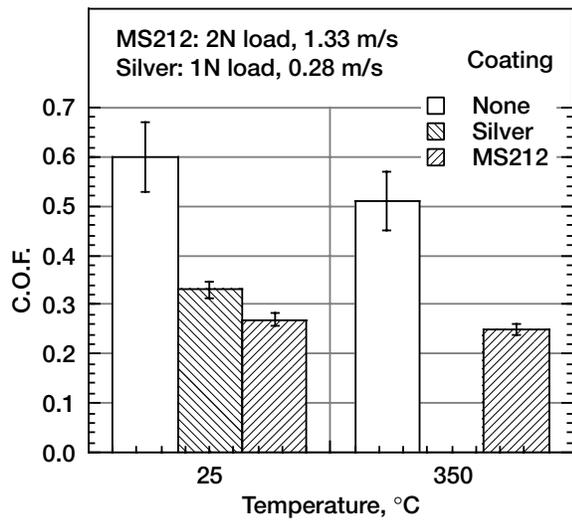


Figure 10.—COF of 6B pins sliding on uncoated and MS212-coated inconel X-750 at room temperature and 350 °C.

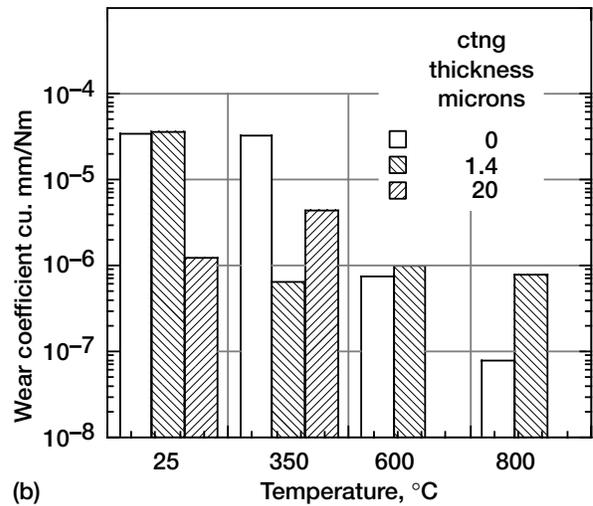
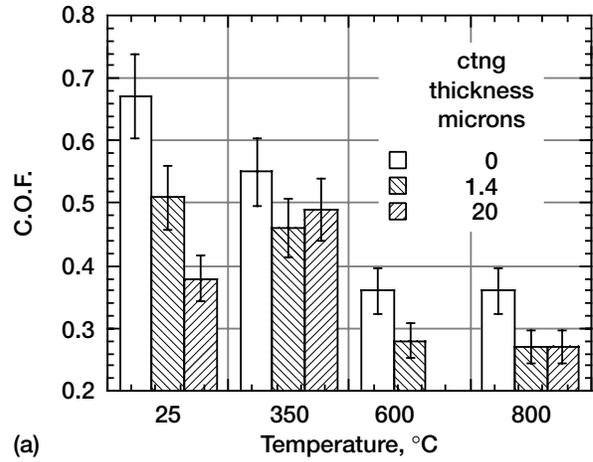


Figure 11.—COF and pin wear for alumina pins sliding on uncoated and MS212-coated inconel X-750 to 800 °C. (a) COF. (b) Pin wear coefficients.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (<i>Leave blank</i>)		2. REPORT DATE May 1997	3. REPORT TYPE AND DATES COVERED Technical Memorandum	
4. TITLE AND SUBTITLE MS212-A Homogeneous Sputtered Solid Lubricant Coating for Use to 800 °C			5. FUNDING NUMBERS WU-243-30-07	
6. AUTHOR(S) Harold E. Sliney, William J. Waters, and Richard Soltis				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135-3191			8. PERFORMING ORGANIZATION REPORT NUMBER E-10699	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001			10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-107437	
11. SUPPLEMENTARY NOTES Harold E. Sliney, Distinguished Research Associate; William J. Waters and Richard Soltis, OMNI Corporation, 21000 Brookpark Rd., Cleveland, Ohio 44135 (work performed under NASA Contract M0065). Responsible person, Harold E. Sliney, organization code 5140, (216) 433-6055.				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Categories 23, 24, and 37 This publication is available from the NASA Center for AeroSpace Information, (301) 621-0390.			12b. DISTRIBUTION CODE	
13. ABSTRACT (<i>Maximum 200 words</i>) Composite coatings containing chromium carbide, stable fluorides and silver were prepared by magnetron sputtering. The microstructure of the coatings is very homogeneous compared to that of plasma sprayed and sintered versions of the same chemical composition. Friction and wear of MS212-coated and baseline uncoated aluminum and Inconel X-750 are compared. At room temperature, the friction and wear of coated aluminum is dramatically better compared to the baseline. The acceptable load is limited by deformation of the soft aluminum substrate. In the case of the nickel alloy, lower friction is observed for the coated alloy at all temperatures up to the maximum test temperature of 800 °C. Pin wear factors for sliding against the coated alloy are lower than the baseline at room temperature and 350 °C, and comparable to baseline wear at higher test temperatures. Low baseline wear at high temperatures is due to the lubricious nature of the natural oxides formed on nickel-chromium alloys in a hot, oxidizing atmosphere. No load limit was found for coated Inconel-X 750 at loads up to five times the load limit for coated aluminum.				
14. SUBJECT TERMS Physical vapor deposited lubricant film; High temperature lubricant coating; Sputtered composite; Tribological coating			15. NUMBER OF PAGES 14	
			16. PRICE CODE A03	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	